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Abstract

The track damage created in LiF crystals by swift U, Xe and Kr ions with a specific energy of 11.1 MeV/u was studied using dislocation mobility measurements, track etching, SEM, AFM and optical microscopy. The results demonstrate high sensitivity of dislocation mobility to track core damage. The relationship between the energy loss of ions, dislocation mobility and track structure is discussed.

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1. Introduction

The use of swift heavy ions has been increasingly of interest for nanostructuring of materials and improving their optical, mechanical and other properties [1]. The investigations of track damage and effects induced by swift heavy ions in solids become active areas of research. Previous studies have revealed the advantages of the dislocation mobility (DM) technique for the track damage diagnostics in LiF [2-5]. The reduction of DM in crystals irradiated with Pb, Bi and U ions producing track core damage was observed above surprisingly low threshold fluence of about 10^6 – 10^7 ions/cm^2 [4-5]. The ion induced increase of indentation hardness manifests at markedly higher fluences (above 10^9 ions/cm^2) [3].

In this work we extend the studies on track core damage in LiF combining the DM, track etching [6] and AFM techniques and using ion species (U, Xe and Kr) with strong difference in energy loss.

2. Experimental

All experiments were performed on nominally pure LiF single crystals (Korth Kristalle, Germany) irradiated with the U and Xe ions of a specific energy of 11.1 MeV/u at the UNILAC (GSI, Darmstadt, Germany) and Kr ions (11.4 MeV/u) at the CIMAP-GANIL (Caen, France) accelerators applying fluences from 10^6 to 10^{11} ions/cm^2. The range of ions and the energy loss were calculated by the SRIM 2006.02 code. The ion tracks and dislocation rosettes produced by Vickers indentation were observed using chemical etching in a saturated aqueous $FeCl_3$ solution. The tracks were etched selectively because their etching rate exceeded that for grown-in dislocations. The distance l between the edge dislocation arms extending along the <110> axes was measured and the relative variation of the arm length expressed as $(l_0 - l)/l_0$, where l_0 is the dislocation arm length in non-irradiated crystal was selected as a parameter characterizing the DM [4]. The measurements were performed on irradiated (001) surface and on cross-sections produced by cleaving crystals perpendicular to the irradiated surface. The depth profiles of dislocation mobility on cross-sections were measured at a constant load of 9.8 mN, at which the size of dislocation rosette (about 17 μm) was small compared to the thickness of irradiated layer (~100μm) and large enough compared to the average distance between tracks (up to 3.5μm at the fluence of 10^7 ions/cm^2). The measurements on irradiated surface were performed at loads in the range of 3–500 mN. The indentation depth was deduced as $h=d/7$, where d is the length of impression diagonal.

3. Results and discussion

3.1 Measurements of dislocation mobility

The DM change in ion-irradiated LiF strongly depends on applied fluence and kind of projectiles (Figs. 1 and 2). The obtained results show that high sensitivity of DM to ion irradiation is observed exclusively for swift heavy ions (U and Xe) exhibiting high energy loss and producing core damage. Similar behaviour was observed in previous studies for irradiations with swift Pb and Bi ions [4, 5]. For this group of projectiles, the ion-induced effect in DM is observed in the range of fluences from 5×10^6 to 10^9 cm^{-2} where the dislocation mobility is reduced mainly by individual tracks. The threshold fluence for different ion species increases with decreasing the energy loss of ions. The observed gradation (Fig.2) we relate to the difference in track core structure.

In contrast to U and Xe ions, the individual tracks of lighter Ni [4] and Kr ions, which consist mainly of single defects in the halo (e.g. F- and H- centres, vacancies and interstitials), cause no detectable change in the mobility of dislocations at low applied fluences. This result demonstrates a negligible role of single colour centres in impeding the dislocation mobility. The reduction of DM is observed at fluences above 10^{10} ions/cm^2 where saturation and aggregation of single defects occurs due to overlapping of the halo regions. The diameter of halo, typically few tens of nm, depends upon the ion energy loss [7, 8].

The depth profiles of ion-induced change in DM for U and Xe ions follow the change of energy loss (Figs. 1(b) and (d)). However, in the case of Kr ions such correlation is observed in a limited range of depth (Fig. 1(f)). The maximum of DM is shifted to the centre of irradiated layer although the maximum damage is expected around the Bragg maximum where the energy loss exceeds the threshold for core damage (about 10 keV/nm [8]). Similar shift was observed for the ion-induced hardening in LiF irradiated with swift Ni ions [9]. The result indicates possible redistribution of core defects by diffusion-controlled post-irradiation processes, which can be facilitated by ion-induced mechanical stresses. Free surface and interface between irradiated and unirradiated parts of the crystal can serve as sinks for mobile radiation defects, such as molecular fluorine. For heavy ions, producing core damage, this effect is observed mainly in the final part of ion path [5] while for lighter Kr ions the discrepancy between DM and energy loss appears in a comparatively wide zone.

For irradiations with U and Xe ions, the change of DM in the near-surface layer is stronger than in the bulk (Figs. 1(a)-(d) and Fig. 2)). The threshold fluence for the measurements on sample cross-sections by about 2 orders of magnitude exceeds that on the irradiated surface. Even low fluence samples with no DM change in the bulk, exhibit significant reduction of DM at the surface. This result is consistent with the previous data for Bi and U ions obtained using indentation hardness and DM techniques [3-5]. The extra-strengthening of surface layer can be related to larger modified area produced by tracks on the irradiated surface. The diameter of highly reactive core region of U tracks revealed on irradiated surface by high-resolution scanning electron microscopy was about 12 nm [10] and surface hillocks observed by scanning force microscopy was about 30 nm [11], while the diameter of track core in the bulk determined by the small angle x-ray scattering was only few nanometres [8]. As a result, the stage of track overlapping and aggregating of single defects on irradiated surface could be reached at lower fluences than in the bulk. Additional surface modification could be caused by post-irradiation processes due to environmental attack and sink nature of the surface for mobile radiation defects.

In contrast to swift heavy ions, the irradiations with lighter Kr ions cause stronger reduction of DM in the bulk (Fig. 2). Obviously, this is due to the fact that the energy loss for Kr ions exceeds the threshold for core damage only at a definite depth around the Bragg maximum.

The investigations of DM revealed some limitations of the method. The etching of dislocation rosettes was successful for low and moderate fluences where the $\Delta l/l_0$ values are below 60-65%. For higher irradiations, the selective etching of indentation-induced dislocations is suppressed. At the lowest fluences, the change in DM was dependent on the applied load, which determines the size of dislocation rosette. No effect was observed at loads where the size of dislocation rosette became comparable with the mean distance between tracks. The saturation of the ion-induced effect in DM on surface was reached when the area of the dislocation rosette included numerous tracks (at least 10-20 tracks of U ions or about 80-100 tracks of Xe ions). For U ions, these conditions were reached at the load of 9.8 mN while the load of 39.2 mN was required for low fluence irradiations with Xe ions.

3.2 Track etching

It is well established that swift heavy ions produce in LiF latent tracks, which can be revealed by etching as sharp-ended pyramidal etch pits [6]. The SEM, AFM and optical microscopy measurements on irradiated surfaces confirm such mode of etching for U ions (Fig. 3(a)). The size of etch pits increases with increasing the etching time confirming the continuity of etching. However, in the case of Xe ions, only about 30% of the etch pits were sharp-ended. Most of etch pits had a flat-bottomed or two-step shape (Fig. 3(b) and (c)). By analogy with the etching of moving dislocations in LiF [12] we consider the flat-bottomed etch pits as evidence for discontinuity of the track etching. Analysis of the height profiles of etch pits shows that the distance between etchable fragments reaches up to hundred nanometres. When the non-etchable interval is passed, the etching of the track is continued as shown in Fig.3(c).

The tracks are etched selectively due to higher chemical reactivity of the track core region and due to elastic stresses created in the surrounding crystal by aggregates of radiation defects. The etching of narrow core region dominates only at early stages of etching [10]. The continuity of etching of U tracks for comparatively long durations gives evidence for the overlapping of stress fields created around neighbouring defect aggregates in track core. Numerous tracks of Xe ions, formed at lower energy loss, exhibit discontinuities in etching, which we consider as a manifestation of non-uniform spacing of defect aggregates in the core region. These results of etching are in agreement with the dislocation mobility measurements, which show that the tracks of U ions are stronger obstacles for dislocations compared to Xe tracks. The discontinuities of etching of Xe tracks are observed in spite of comparatively high energy loss exceeding the threshold of for track etching. Similar effect was observed earlier at the final part of Pb and Bi tracks in LiF [5] where only a part of tracks were revealed by etching and numerous etch pits had a flat-bottomed shape. As shown for some crystals, such as InP the discontinuities of etching can appear due to change of ion charge which leads to oscillations of the energy loss below the etching threshold [13].

4. Conclusions

The results show high sensitivity of dislocation mobility to track core damage. In agreement with earlier results [4] the ion induced reduction of DM on irradiated surface is stronger compared to that in the bulk of irradiated layer. The efficiency of dislocation impeding by tracks decreases with decreasing the energy loss in the row of U$\rightarrow$ Xe$\rightarrow$ Kr projectiles. For U tracks, continuity of etching and high obstacle strength for dislocations is observed while the tracks of Xe ions show discontinuities of etching and reduced obstacle strength. The comparison of the depth profiles of DM and energy loss for Kr ions allows us to suggest possible redistribution of radiation defects by their migration to irradiated surface and interface between irradiated and unirradiated parts of the crystal subjected to stress gradients.

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Figure captions

Fig.1 The relative variation of the dislocation arm length on LiF surface irradiated with U, Xe and Kr ions as a function of indentation depth ((a), (c) and (e)) and the depth profiles of the ion induced change of dislocation arm length and energy loss ((b), (d) and (f)).

Fig.2. The relative variation of the dislocation arm length in LiF irradiated with U, Xe, Kr and Ni ions as a function of fluence. Open symbols denote the measurements on irradiated surface and solid symbols – on the sample cross-sections. The load was 9.8 mN with the exception of 39.2 mN for surface measurements in the case of Xe ions.

Fig.3. AFM images ($1.2\times1.2\ \mu m^2$), height profiles of etched U (a) and Xe (b and c) tracks and schematic of the track structure.

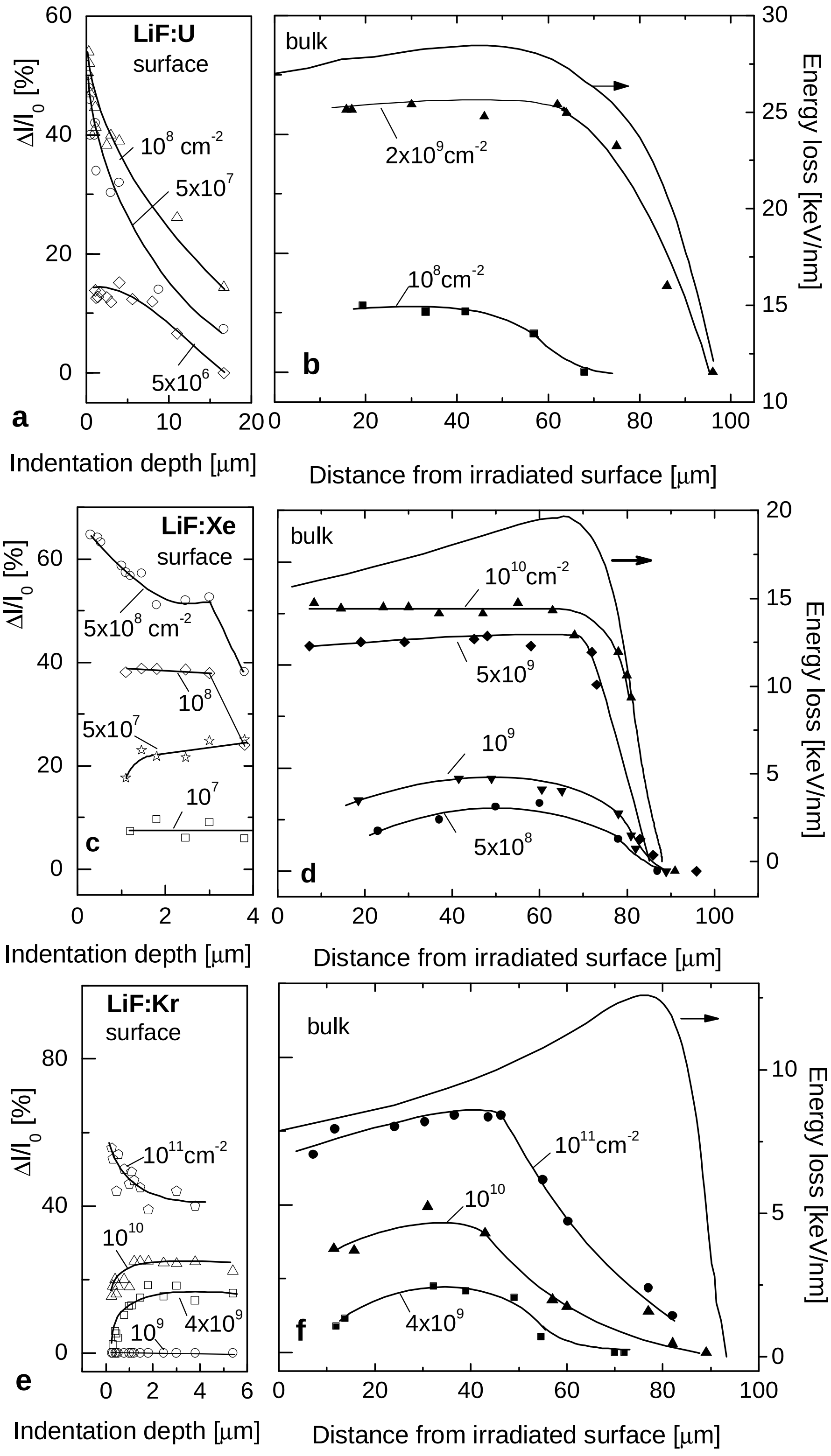

Fig.1.

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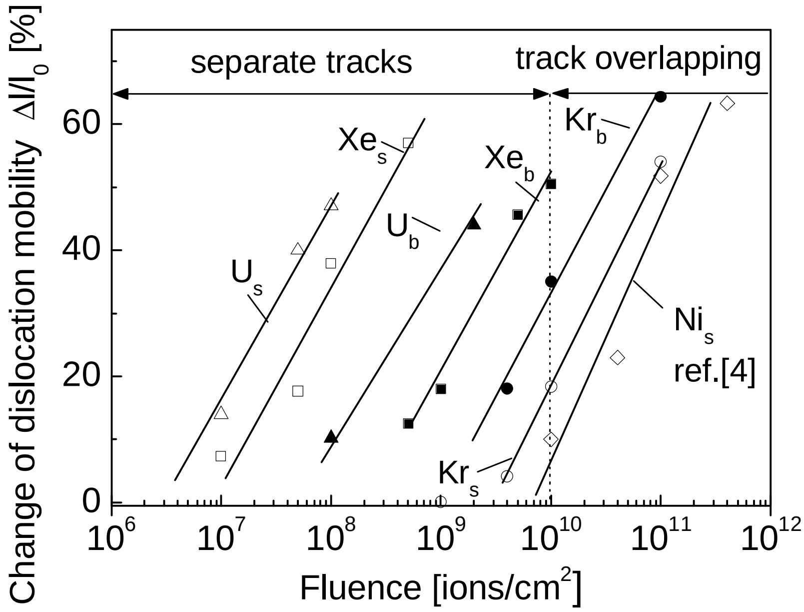

Fig.2

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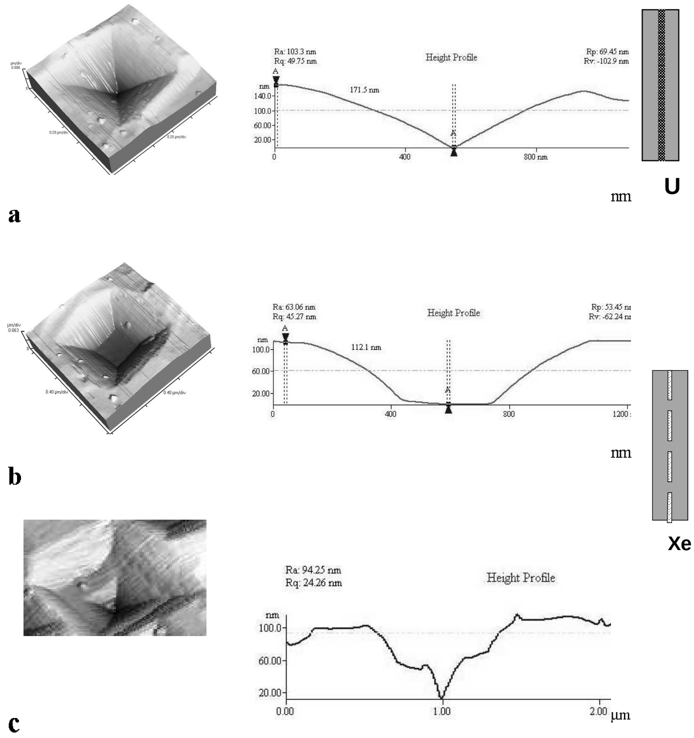

Fig.3

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